PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 3 : G01N 21/63

A1

(11) International Publication Number: WO 83/ 03303

(43) International Publication Date: 29 September 1983 (29.09.83)

(21) International Application Number: PCT/FI83/00023

(22) International Filing Date: 14 March 1983 (14.03.83)

(31) Priority Application Number: 820884

(32) Priority Date: 15 March 1982 (15.03.82)

(33) Priority Country: FI

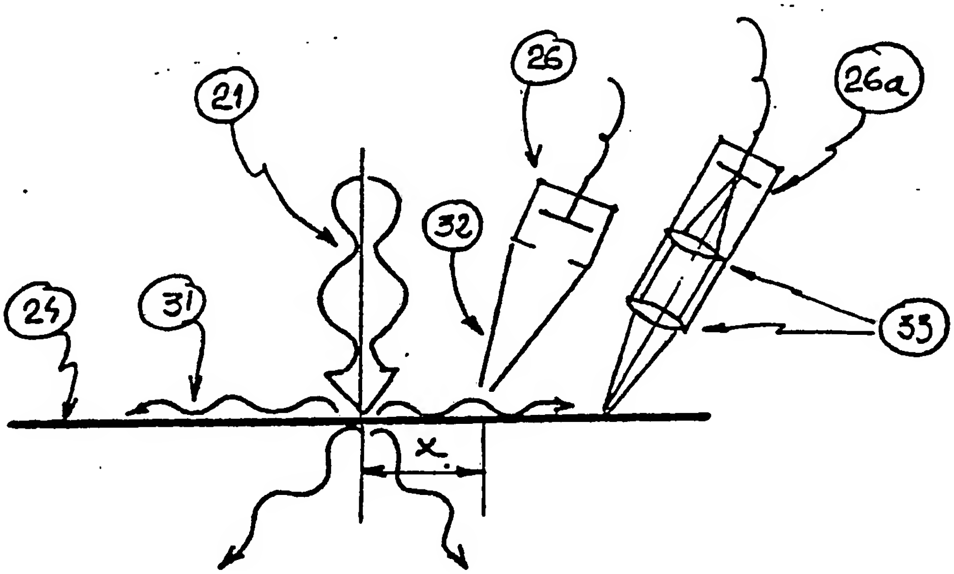
(71)(72) Applicants and Inventors: LUUKKALA, Mauri [FI/FI]; Haukilahdenranta 23 B 5, SF-02170 Espoo 17 (FI). LEHTO, Ari [FI/FI]; Raekuja 3, SF-00700 Helsinki 70 (FI).

(74) Agent: FORSSÉN & SALOMAA OY; Uudenmaankatu 40 A, SF-00120 Helsinki 12 (FI).

(81) Designated States: DE, GB, JP, SE, US.

Published
With international search report.
In English translation (filed in Finnish).

(54) Title: PROCEDURE AND MEANS FOR EXAMINING THE SURFACE QUALITY OF MATERIALS IN SOLID STATE



(57) Abstract

A procedure and a means by which can be measured the characteristics of various solid materials, in particular of surfaces of and coatings on metals and thickness of surface hardening layers without damaging the specimen (24) itself. The procedure is based on measurement of the phase angle of a thermal surface wave (31) produced by a modulated light beam (21). The phase angle of the continuous thermal wave (31) produced by the light energy and progressing along the surface is measured with temperature detectors (26, 26a) either at a fixed distance from the light spot as a function of frequency or at a fixed frequency as a function of distance..Since the phase angle of the thermal wave (31) depends on the thickness of the surface hardening layer, the thickness of the hardened layer can hereby be measured.

AA

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	LI	Liechtenstein
AU	Australia	LK	Sri Lanka
BE	Belgium	LU	Luxembourg
BR	Brazil	MC	Monaco
CF	Central African Republic	MG	Madagascar
CG	Congo	MR	Mauritania
CH	Switzerland	MW	Malawi
CM	Cameroon	NL	Netherlands
DE	Germany, Federal Republic of	NO	Norway
DK	Denmark	RO	Romania
FI	Finland	SE	Sweden
FR	France	SN	Senegal
GA	Gabon	SU	Soviet Union
GB	United Kingdom	TD	Chad
HU	Hungary	TG	Togo
JP	Japan	US	United States of America
KP	Democratic People's Republic of Korea		

Procedure and means for examining the surface quality of materials in solid state

The present invention concerns a procedure and a means for examining the surface defects of materials in solid state, such as metals or equivalent, and the properties of their coatings, such as adhesion, without destruction of the material using a light beam. One specific object of the invention is measurement of the thickness of the surface-hardened layer of steel without contacting the specimen. The procedure and means may also be used in measuring other types of coating, for instance so-called plasma coatings. In the interest of perspicuity, however, the following disclosure is concentrated specifically on coatings upon metals and particularly on surface hardening.

Examination of coatings and of the surface hardening of steel is usually carried out in that a piece is cut from the object to be examined and the structure of the surface, or the thickness of the coating, is examined from one side with an optical microscope. If it is desired to examine the distribution of hardness in the surface, the usual procedure is to press a pointed diamond stylus into the surface of the specimen with constant force and to measure the depth of the resulting depression. The hardness is the higher, the smaller the depression. (This type of instrument is called a Vickers hardness tester.) Similarly, it is also possible to measure e.g. the thickness and other characteristics of paint coats from the side. In case the object to be examined is precious, it is most awkward to cut off specimens.

The aims of the invention are achieved, for instance, by making use of the so-called photothermic effect. If a light beam is directed on the surface of the specimen, part of the light energy is absorbed by the specimen surface and converted into heat. If the light is intensity-modulated at a given frequency, the heating will also be periodic with same frequency. If the intensity of the

incident light is represented by the equation $I = I_o/2\ (1 + \cos\omega t)$ (where $I_o/2 \cos\omega t$ represents the alternating component of the light intensity), then also the temperature at the lighted spot conforms to the equation

$$T = \frac{T_o}{2}\ (1 + A(\omega)\cdot\cos(\omega t+\phi)).$$

Here, $T_o/2$ represents the static rise of temperature on the surface and $T_o/2\ A(\omega)\ \cos\ (\omega t+\phi)$, the alternating component, which displays a certain phase shift ϕ with reference to the light. $A(\omega)$ is the amplitude factor of the temperature, dependent on frequency. This periodic heating of the surface is propagated along the surface of the specimen, and into it, as a kind of "heat wave" or "thermal wave". This thermal wave has a given wavelength and phase angle, the phase changing as a function of time and location. The depth of penetration of this thermal wave is the so-called thermal diffusion length $\mu = \sqrt{\frac{2\alpha}{\omega}}$, with ω the angular frequency of modulation of the light and the thermal diffusivity of the material in question. The penetration of the thermal wave into the material can be expressed by the formula

$$T = T_o e^{-\sqrt{\frac{\omega}{2\alpha}}\cdot x} \cdot \sin(\omega t - \sqrt{\frac{\omega}{2\alpha}} \cdot x)$$

where x stands for the distance which the wave has travelled. It is seen that the thermal wave is exponentially attenuated and that the associated phase angle ϕ has the form

$$\phi = \sqrt{\frac{\omega}{2\alpha}} \cdot x$$

It is important to observe that although the thermal wave penetrates into the material, part of it still proceeds along the surface of the object in the plane of the surface. We shall in this invention specifically use this continuous wave propagated along the surface, and we measure its phase angle as a function of loca-

tion x or as a function of ω with an infra-red detector or with a thermocouple. This is done because it can be shown that the wave progressing along the surface is quite sensitive specifically to the properties of the surface and to the quality of various coatings.

It is highly important to note that the phase difference is measured at least at two different values of x or at least at two different values of ω. This enables the fixed phase differences present in the apparatus to be eliminated and the true thermal diffusivity of the surface to be estimated. In any other procedure an incorrect value would be obtained for the thermal diffusivity. The present invention differs in this respect from all apparatus and procedures known in the art.

In the procedure and apparatus of the Finnish patent application No. 801850 is also used a light source (a laser) modulated at a given frequency and an infra-red meter recording the periodic increase of the surface temperature. This apparatus operates well enough in itself and various kinds of measurements on coatings can be carried out with it. It has been found, however, that the sensitivity of this apparatus is fairly low particularly in surface hardening measurements. In the apparatus of said patent application No. 801850, the periodic variation of surface temperature is observed over a comparatively extensive area. Because the periodic temperature increase is highest specifically at the point where the laser beam strikes, the apparatus mentioned will measure the properties of the specimen surface mainly at the location of the light spot only. In the present invention, the amplitude and phase angle of the thermal wave are specifically measured at a great enough distance from the point heated by the light. When the measurement is made of the phase angle of the continuous thermal wave progressing close to the surface or along the surface, it is naturally most sensitive exactly to the properties of the surface. Since it is possible to measure the phase angle without contact with an infra-red detector, no load whatsoever is imposed on the propagation characteristics of the thermal surface wave. It is possible at the same time to measure the behaviour of the phase angle as a

function of distance x by displacing the detector gradually away from the heating light spot. If the diffusivity of the specimen surface is α_p, the phase angle ϕ of the thermal wave progressing along the surface at a given frequency ω is also found by the formula

$$\phi = \sqrt{\frac{\omega}{2\alpha_p}} \cdot x$$

If the contactless infra-red detector has been connected to a phase detector where the phase of the measured signal is compared with a fixed reference signal having the same frequency ω, it is easy from the output of the phase detector to calculate the diffusivity α_p of the surface because x is known. The diffusivity α_p is dependent on the quality of the coating, and in the case of surface hardening for instance it is dependent on the thickness of the surface-hardened layer. It is likewise possible to estimate the thickness and adhesivity e.g. of paint coats or of so-called plasma layers.

The present procedure and apparatus have the further advantage that since the angular frequency of modulation ω can be regulated, it is possible at the same time to control the depth of penetration of the thermal wave and thereby the accuracy of measurement in the case of thin coatings. Two different techniques for measuring diffusivity can be applied with the practical measuring apparatus: the measuring distance x from the light spot is kept constant and is adjusted, or ω is kept constant and the phase angle is measured for several different values of x. It is believed that the second mode is easier to carry out in practice.

Another special feature of the present invention is that the phase angle of the thermal wave progressing along the surface is measured by means of temperature pick-ups where the metallic specimen itself constitutes a component of the thermocouple. It is known from basic physics that if a junction is formed of two different metals and this junction is heated, an electromotive force builds up across the junction which is dependent on the temperature. In the present

invention, for instance the tip of a tungsten wire is pressed against the surface of the metallic specimen and the voltage produced between the specimen and the tungsten wire is measured with an electronic measuring instrument. Hereby, when heating up, the specimen forms a thermocouple with the tip of the tungsten wire and the thermal load caused by the wire will be minimal. It is now possible by moving the light spot and the measuring tip with reference to each other to change the distance x between them, to determine the phase angle of the thermal surface wave in the manner already described.

Another arrangement is that in which against the specimen are pressed two closely spaced metal tips consisting of such metal that they form a thermocouple with the specimen itself. The advantage of this arrangement is that for measuring the generated voltage no particular electrical contact need be provided with the specimen, instead of which the voltage between the two tips constituting the thermocouple is measured. If the tips are spaced by a given, appropriate distance, the phase angle of the thermal surface wave is immediately obtained from the phase difference between the voltages produced by these two thermocouples.

The arrangement described above has the advantage that the set-up is not highly critical as regards the quality of contact between the tip and the specimen, since in the measurement specifically the phase angle is observed, which is independent of the amplitude of the electrical signal itself. On the other hand, the arrangement just described is independent of variations which may occur in the ambient lighting and of diffuse light scattered by the specimen surface. Thirdly, the specimen itself serves as one component of the thermoelectric couple, whereby no thermal loading effect imposed on the surface by a conventional thermocouple to be mounted separately is incurred.

In experiments carried out in practice, much stronger signals have been obtained with this thermocouple arrangement than with infra-red detectors, the sensitivity to interference being reduced

at the same time. It is to be noted, on the other hand, that infra-red detectors may be used to measure the properties of insulating materials as well.

As taught by U.S. Patent No. 3.222.917, defects, cracks etc. can be measured in objects in that in the specimen is produced a single local thermal pulse. This thermal pulse is then detected, for instance, by means of thermocouples or equivalent pressed against the surface. It is then possible by observing the time of ascent, the time of descent and the shape of the heat pulse derived from these thermocouples to draw conclusions concerning the properties of the specimen. The present invention does not employ a single pulse and its deformation: it uses continuously intensity-modulated light for source of a thermal wave and measures the behaviour of the phase angle of this continuous wave. It is moreover pointed out that for a single pulse no phase angle can be defined: the phase angle is exclusively a characteristic of a continuous wave. Another decisively important difference is that since the modulation frequency ω can be controlled, it is also possible to control the depth of penetration of the thermal wave. The single-pulse arrangement does not afford this possibility. Since in this invention we are specifically interested in coatings, we have to be able to control the depth of penetration by means of an appropriate ω, because it should be remembered that the depth of penetration is inversely proportional to the square root of ω. It should further be noted that if temperature pick-ups are pressed against the surface, they may alter the propagation of the thermal pulse on the surface. This is why in the present invention are used contactless infra-red meters or a thermocouple where the specimen itself is a component of the thermocouple.

The U.S. Patent No. 3.043.956 discloses an arrangement wherein the specimen is irradiated by a modulated infra-red source and is then observed with the aid of infra-red detectors, and the phase of the resulting signal is observed with reference to the phase of the modulation of the radiation source. In the present invention, firstly, no infra-red light source is used like in said U.S.

patent. Secondly, in the present application is specifically studied the wave progressing along the surface, and its phase, as a function of the distance travelled across the surface. The measurement itself is made specifically at a point different from that on which the incident radiation is directed, and the use as parameter of the distance x described above is essentially important with a view to measuring the quality of the coating and the thermal diffusivity of the surface. In the said U.S. patent an arrangement is encountered wherein the infra-red source and the detector have been mounted on one and the same side of the specimen with a given mutual spacing, but this arrangement has been chosen in order to compensate for the movement of the specimen. This arrangement also fails to vary the distance x in the direction of the surface for measurement of phase differences.

The present invention is characterized by the feature stated in the claims.

In Fig. 1 is schematically illustrated the generation and propagation of the thermal surface wave.

Fig. 2 illustrates in greater detail the construction principle of the apparatus of the present patent application.

In Fig. 3 is presented the arrangement for measuring phase differences with the aid of metallic points.

In Fig. 4 is presented the measuring of phase differences with the aid of two points.

As shown in Fig. 1, a light beam 21 intensity-modulated at a given frequency periodically heats the desired point on the surface of the specimen 24. This heated point emits a thermal wave 31 in all directions in the specimen 24, and part of the thermal energy proceeds as a wave 31 along the surface of the specimen with a given velocity and a given phase angle dependent on location and on the thermal diffusivity of the surface. The wave 31 proceeding on

the surface is observed by means of one or several infra-red detectors 26 or 26a mounted in a suitable manner. The aperture of this infra-red detector has been limited so that it monitors only a small part of the specimen's surface so that the direct thermal radiation of the scattered visibly light radiation from the illuminated point does not strike the detector. This aperture limiting can be accomplished for instance by means of a trumpet-like structure 32 or an infra-red lens assembly 33. The measuring distance of the infra-red detector from the illuminated point can be adjusted e.g. by mechanical arrangements in the direction of the surface. It should be noted moreover that the detector is AC-connected, in other words, it only observes the alternating signal but not the static temperature rise in the specimen.

In Fig. 2 is schematically depicted the apparatus as a whole. The light beam 21 coming from the light source 20, usually a laser, passes through a light intensity modulator 22 operating at an adjustable frequency, to the focussing lens 23, the specimen 24 being mounted in the focal plane of the latter. If desired, the specimen may be moved in the focal plane by means of an electrically controlled manipulator 25. The thermal surface wave 31 emitted by the specimen surface heated by the light beam 21 is observed with one or several infra-red detectors 26 mounted far enough from the light spot, the detector's aperture being limited in a convenient way so that it monitors only a small enough part of the specimen surface. The distance of the infra-red detector from the light spot in the direction of the surface can be controlled as desired by the aid of an electrical manipulator 27. The signal from the infra-red detector is conducted to a phase-sensitive detector 28, to which is simultaneously conducted the fixed reference signal 29 from the light modulator 22. The output signal from the phase-sensitive detector is carried to a suitable output device 30, which may be e.g. a recorder or a microprocessor. The microprocessor may control the manipulator 27 so that an output record is obtained of the phase angle as a function of the distance x. It is obvious that if the specimen surface is heated at one single point, the thermal wave on the surface of the specimen will start to progress

radially. The aperture of the infra-red detector may then be limited for instance with a trumpet structure shaped like a half-circle. If the heating is done e.g. in band fashion, the aperture may consist of a slit-like trumpet structure. A slit-like aperture may also be obtained with a cylindrical lens. The lenses should in that case be germanium lenses, which are transparent to infra-red rays.

In Fig. 3 is depicted a measuring set-up in which the metallic specimen itself is part of a thermocouple. The phase angle of the thermal surface wave 31 progressing along the surface of the metallic specimen 24 is measured by the aid of a metallic measuring point 40 pressed against the surface. The material of the point is so selected that the junction 41 with the metallic specimen constitutes a thermocouple. The electromotive force, dependent on temperature, generated in this thermocouple is measured with the phase meter 28 as a function of the distance x between the light spot and the measuring point with at least two different values of x. In order to accomplish a closed circuit, the specimen 24 has to be connected to the phase detector by a lead 42. It is also necessary to conduct to the phase detector a reference signal from the light modulator in accordance with the well-known principles of phase measurement. The distance x is controlled as in the arrangement shown before in Fig. 2.

In Fig. 4 is depicted an arrangement in which the phase difference is measured with two measuring points 40 and 40a, the metallic specimen 24 being part of the thermocouple in them. A closed circuit is here established through both measuring points, whereby no separate lead is required as in the case of Fig. 3. Since the measurement takes place at two different points, it is not absolutely necessary to adjust the distance between the light spot and the points of measurement and one single measurement is sufficient to elicit the correct phase difference.

The materials of the measuring points have to be of such substance that a thermoelectric voltage is generated between the measuring

point and the metallic specimen. It has been established by experiments that for instance a measuring point made of tungsten yields a good signal. Also measuring points made of sintered materials produce excellent signals.

In the foregoing has only been presented the principle of the invention and one advantageous embodiment thereof. It is obvious to a person skilled in the art that the invention may be modified in various ways within the inventive idea presented in the claims.

Claims

1. Procedure for measuring the thermal diffusivity of solid materials, such as metals, coatings and surface layers and the thickness and other characteristics of a surface hardening layer, characterized in that in the surface of the body under examination is produced by means of a light beam intensity-modulated at desired frequency a continuous, progressing heat wave, of which the phase angle is measured in the direction of the surface either as a function of the distance in the direction of the surface or as a function of the modulation frequency, with infra-red thermometers or with a thermoelectric couple in which the specimen itself is part of the thermoelectric couple.

2. Means based on a procedure according to claim 1 for measuring the thickness and other characteristics of solid materials, such as coatings on and surface layers of metals and surface hardening layers, characterized in that on the surface of the specimen (24) under examination is directed from a light source (20) a light beam (21) modulated at desired frequency by an intensity modulator (22), said light beam heating the surface of the specimen periodically so that in the surface of the specimen is produced a continuous and progressing thermal wave (31), of which the phase angle is measured as function of the distance in the direction of the surface with one or several infra-red detectors (26) so that the electronic signal from the infra-red detectors is carried to a phase detector (28), to which is also conducted a reference signal (29) with a fixed phase from the light intensity modulator (22).

3. Means according to claim 2 measuring the characteristics of surface layers, characterized in that the phase angle of a continuous thermal wave progressing along the surface is measured with infra-red detectors (26) not contacting the surface so that the distance of the point of measurement from the light spot in the direction of the surface is constant but the phase angle of the thermal wave is measured as a function of the frequency of the light intensity modulation.

4. Means according to claim 2 or 3 measuring the characteristics of surface layers, characterized in that the measuring aperture of the infra-red detectors (26) has been limited either with a trumpet-like structure (32) or with an infra-red lens assembly (33) to be either band-like or shaped like a half-circle, depending on whether a light band or a light spot, respectively, is formed with a focussing lens (23) on the surface of the specimen (24) in the focal plane to constitute the source of the thermal wave.

5. Means according to claim 2 or 3 measuring the characteristics of surface layers of metallic specimens, characterized in that the phase angle of the continuous thermal wave (31) progressing along the surface is measured as a function of the distance in the direction of the surface with one or several measuring points (40) touching the surface and constituting a thermoelectric couple together with the specimen (24) itself so that the electronic signal from the circuit constituted by the measuring points and the specimen is carried to a phase detector (28), to which is also conducted a reference signal (29) from the light intensity modulator.

6. Means according to claim 2,3 or 5 measuring the characteristics of surface layers of metallic specimens, characterized in that the phase angle, in the direction of the surface, of the continuous thermal wave (31) progressing along the surface is measured with two measuring points (40,40a) touching the surface and constituting a thermoelectric couple and a closed circuit with the specimen (24) itself and located at two different distances from the light spot.

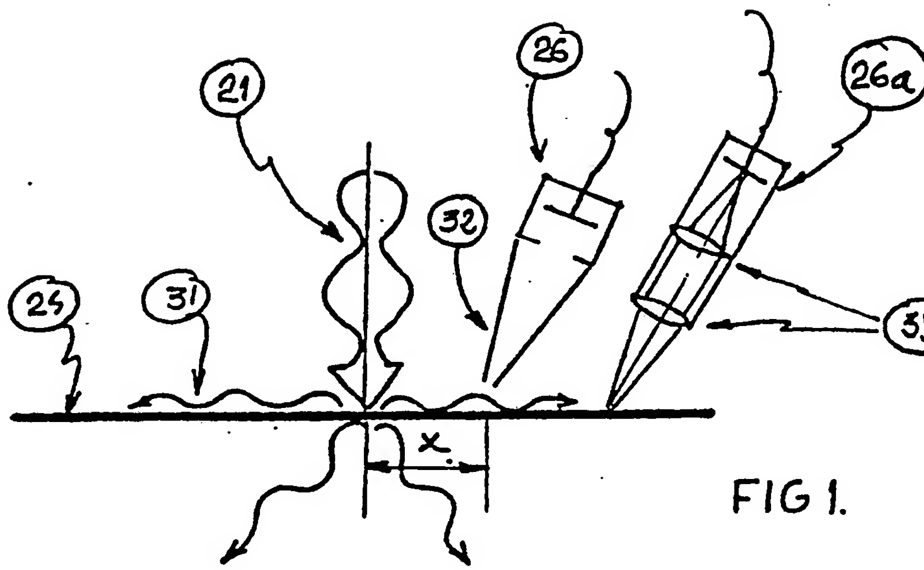


FIG 1.

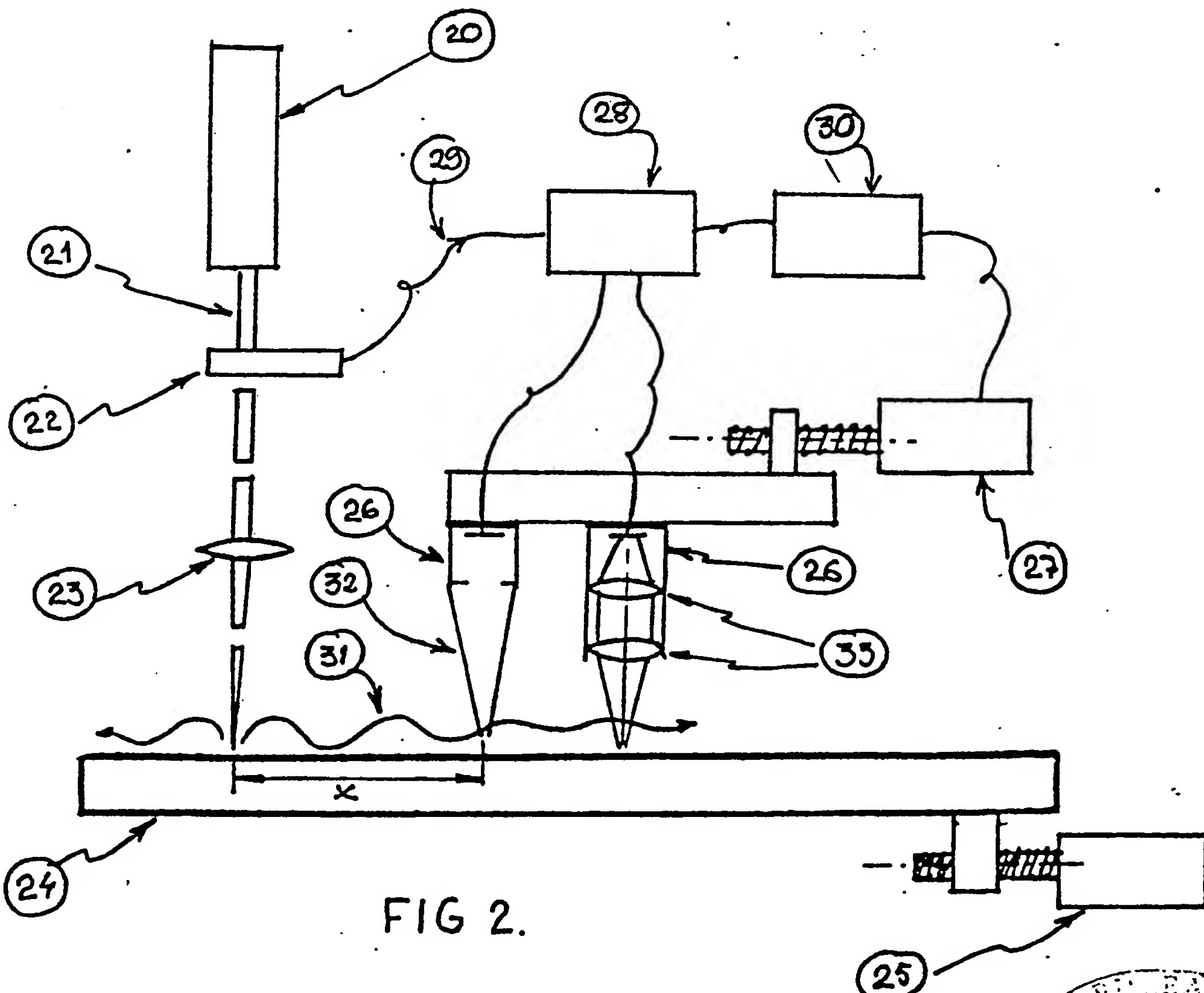


FIG 2.

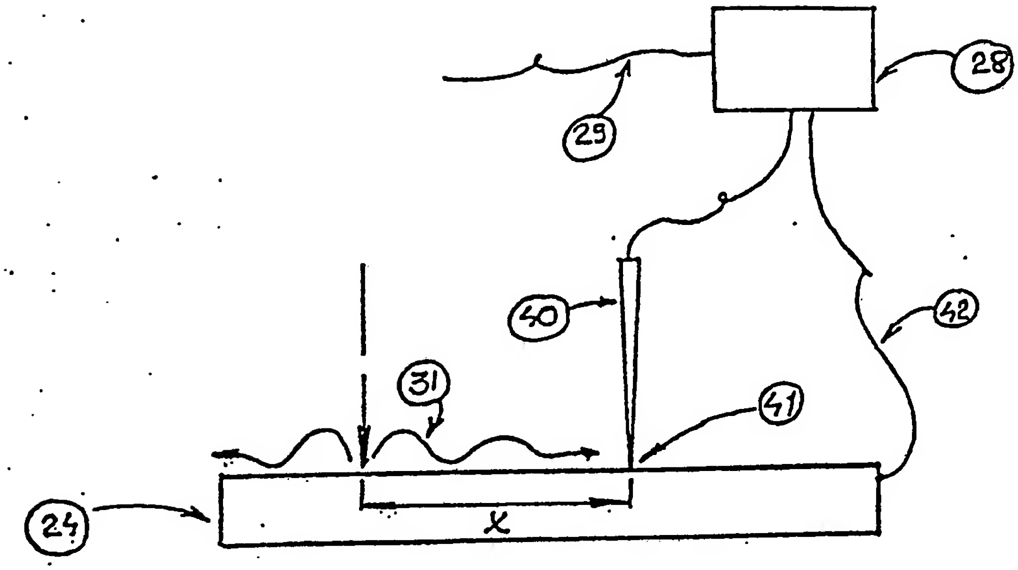


FIG 3.

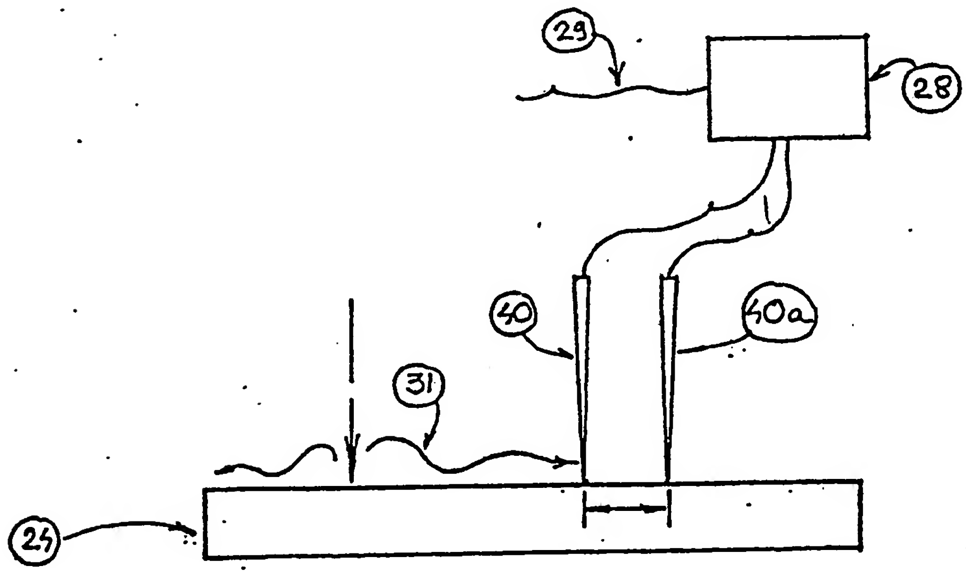


FIG 4.

INTERNATIONAL SEARCH REPORT

International Application No PCT/FI83/00023

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [3]

According to International Patent Classification (IPC) or to both National Classification and IPC 3

G 01 N 21/63

II. FIELDS SEARCHED

Minimum Documentation Searched [4]

Classification System	Classification Symbols
IPC 3.	G 01 N 21/35,49,62-65,71,86, 25/18, 25/72 G 01 B 11/06,30, G 01 J 5/58, G 01 S 17/74, C 25 D 21/12

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [5]

SE, NO, DK, FI classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT [14]

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No. [18]
Y	WO, A1, 81/03704 (VALMET OY) 24 December 1981	1
Y	GB, A, 1 403 950 (COMMISSARIAT A L'ENERGIE ATOMIQUE) 28 August 1975	1
A	US, A, 3 043 956 (FRANKLIN SYSTEMS INC) 10 July 1962	1-6
A	US, A, 3 222 917 (GABB SPECIAL PRODUCTS INC) 14 December 1965	1-6
A	DE, A1, 2 502 289 (NATIONAL RESEARCH DEVELOPMENT CORP) 12 November 1975	1-6

* Special categories of cited documents: [15]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search [2]	Date of Mailing of this International Search Report [2]
1983-05-09	1983 -05- 20
International Searching Authority [1]	**Signature of Authorized Officer [20]**
Swedish Patent Office	Monica Eurenius Monica Eurenius

International Application No. PCT/FI83/00023

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

II

Fields Searched (cont)

IPC 2 G 01 N 21/32,34,36,
G 01 B 19/38,60

National Cl 42b:12/02; 42k:46/07

US Cl 250:83.3, 219;
356:51, 120, 156, 167, 201,
237, 371, 381, 432, 435

V.☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [10]

This International search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1.☐ Claim numbers .... because they relate to subject matter [12] not required to be searched by this Authority, namely:

2.☐ Claim numbers ...., because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out [13], specifically:

VI.☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [11]

This International Searching Authority found multiple inventions in this international application as follows:

1.☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2.☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3.☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4.☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.